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EVALUATION OF REPLACEMENT RED SMOKE DYES
FOR 1-N METHYLAMINOANTHRAQUINONE

by

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Munitions Division

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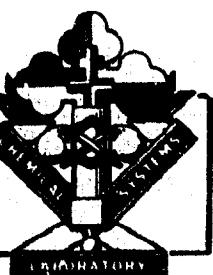
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <i>(21)</i> A list of 114 dyes was compiled from a literature search for replacement red smoke dyes for 1-N methylaminoanthraquinone. Two of the dyes reviewed (9-diethylaminorosindone and O-methoxyphenylazo-B-naphthol) were found to meet the requirements for use in pyrotechnics. There are insufficient toxicological data at this time to assess the health effects of these dyes.		

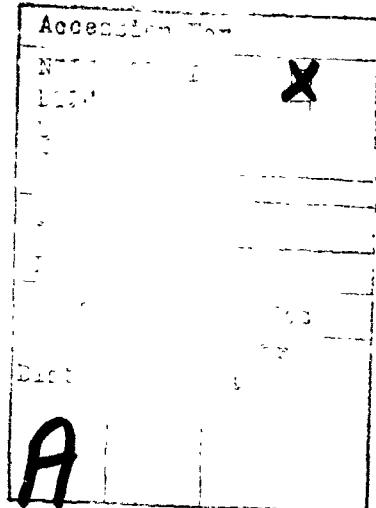
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PREFACE

The work described in this report was authorized under Project 1A98772, Engineering Study to Determine Feasibility of Replacing Dye, Disperse Red 9. This work was started in November 1979 and completed in May 1980.

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EVALUATION OF REPLACEMENT RED SMOKE DYES FOR 1-N METHYLAMINOANTHRAQUINONE

1. INTRODUCTION

The objective of this work was to compile a list of possible replacement compounds for the dye currently employed to generate red smoke by pyrotechnic methods. The red dye now used is 1-N methylaminoanthraquinone.

2. METHOD

A list of dyes used or tested for use in pyrotechnics was compiled by reviewing open literature and reports available in the Technical Library of Chemical Systems Laboratory.

Representatives from dye manufacturing companies were consulted to determine whether any new available dyes could be employed for use with pyrotechnic compositions.

The list of Food and Drug Administration dyes subject to certification (revised April 1, 1979) was reviewed for replacement dyes. These dyes are commercially available, and toxicological testing has already been performed on each.

3. RESULTS

Table 1 lists the dyes from the open literature and from reports in the Technical Library which have been used or tested for use in pyrotechnic smoke compositions.

Included with the list of dyes is the following:

- a. Manufacturer, if available.
- b. Other names - commercial or chemical.
- c. Colour Index (CI) number (Numbers before 10000 are no longer listed in the current Colour Index.)

Table 2 lists the Food and Drug Administration dyes reviewed. Current Colour Index numbers are noted.

Consultations with dye manufacturers yielded one possible dye, Rhodamine B.

Table 1. Dyes Used or Tested for use in Pyrotechnics

Dye		Reference
1. Acetanilide Scarlet B	Made by DuPont	1, 3
2. AD 687		3
3. AD 722		3
4. AD 779		2
5. Alpha-amino anthraquinone		1, 4, 5
6. Amino azobenzene		3
7. Amino azotoluene- β -naphthol	Celanthrene Red 3B Made by DuPont	1, 3, 4
8. 2-Amino biphenyl- β -naphthol		3
9. 1-Amino-4-hydroxy anthraquinone		6
10. Aniline-1-hydroxy naphthalene-4-sulfonic acid		1
11. Aniline azo- α -naphthol	Rosinduline GX, Cl 828	3
12. 5-Anilinobenzo (a) phenazine		6
13. o-Anisidine-1-hydroxy naphthalene-4-sulfonic acid		1
14. p-Anisidine-1-hydroxy naphthalene-4-sulfonic acid		3
15. o-Anisidine azo α -naphthol		3
16. o-Anisidine azo resorcinol		6
17. 4-Anisole azo β -naphthol		6
18. Azo Red K 1858	Fast Blue BB	2
19. 1-(4-Benzamido-2,5-dimethoxy phenylazo)-2-naphthol	Dry Red X9848	7
20. 1-(2-Biphenylazo)-2-naphthol	Made by Calco	1
21. Brilliant Red R	Ciba Violet B, Cl 1222	1
22. 5-Bromo-2-(5,7-dibromo-3-oxo-3(2)-indolydine)-3(2)-thianaphthene	*Cl 73620 (Vat Dye) 8	7
23. Calcolake Red D		1, 3
24. p-Chloroaniline- β -naphthol		3
25. p-Chloro-o-anisidine-1-hydroxynaphthalene-4-sulfonic acid		2, 3
26. 4-Chlor-2-anisole-azo- β -naphthol		1, 2, 3
27. 4-Chlor-2,5-dimethoxybenzene-azo- β -naphthol	Calcolake Red C	1
28. 5-Chloro-2-(hydroxy-1-naphthylazo)-p-toluene sulfonic acid	Sulfanthrene Red 3B	1
29. 5,5'-Chloro-6,6'-methylthioindigo	Cl 1212	1
30. 4-(p-Chlorophenylazo)-N,N-dihydroxy ethylaniline		1
31. 4-(p-Chlorophenylazo)-N-ethyl-N-(2-hydroxyethyl) aniline		1
32. Cresidine-1-hydroxy naphthalene-4-sulfonic acid		3
33. 1,5-Diamino anthraquinone		4
34. 1,4-Dibenzoylamino anthraquinone		1
35. 2,4-Dichloroaniline- β -naphthol		4
36. 2,5-Dichloroaniline- β -naphthol		1, 3
37. 2,5-Diethoxyaniline- β -naphthol		1, 3
38. 9-Diethylamino rosindone	9-Diethylamino-7-phenyl-5-benzo (a) phenazinone (MIL-D-3614)	1, 2, 5, 7
39. 9-Diethylamino-7-p-tolyl-5-benzo (a) phenazinone	Rosindone National AD	9-11
40. N,N-Diethyl-p,p'-nitrophenylazocanine		1, 7, 9
41. 2,5-Dimethoxybenzene-azo- β -naphthol		1
42. 1,5-Dimethylamino anthraquinone	*Cl 12156 (Solvent Red 80) 8	2, 3
43. 1,8-Dimethyl-3,6-dichloro thioindigo		6
44. 4-(2,4-Dinitro phenylazo) phenol		3
45. 6-Ethoxy-2-amino-benzthiazole-ethylanilol		1
46. 5-Ethyl-2-anisole-azo- β -naphthol		1, 3
47. Federal Continental Red		2
		3

* See footnote at end of table.

Table 1. (Contd)

Dye		Reference
48	Federal Independence Red	3
49	Federal Lincoln Red	1, 3
50	Federal Signal Red B	3, 7
51	Federal Smoke Red D	7
52	4-Hydroxy-3-(2,4-dimethoxy phenylazo)-1-naphthalene sulfonic acid	1
53	4-Hydroxy-3-(p-ethoxyphenylazo)-1-naphthalene sulfonic acid	1
54	4-Hydroxy-3-(2-hydroxy-m-tolylazo)-1-naphthalene sulfonic acid	1
55	4-Hydroxy-3-(o-methoxy-p-chlorophenylazo)-1-naphthalene sulfonic acid	1
56	4-Hydroxy-3-(o-methoxyphenylazo)-1-naphthalene sulfonic acid	1
57	2-(2-Hydroxy-1-naphthylazo)-1-naphthalene sulfonic acid	1
58	4-Hydroxy-3-phenylazo-1-naphthalene sulfonic acid	1
59	1-(2-Hydroxy-m-tolylazo)-2-naphthol	1
60.	Indophenol BG	1
61	Indophenol RL	1
62	Induline Base 5G	1
63	Isorosinduline	3
64	K1840 (see references 3 and 14)	7
65	Lithol Red Toner	7
66	1-(4-Methoxy anilino) phenylazo)-2-naphthol	1
67.	1-(2-Methoxy-4-nitrophenylazo)-2-naphthol	1
68	o-Methoxyphenylazo- β -naphthol	1, 2, 3, 5, 6, 9, 12
69	5-Methyl-2-anisole-azo- β -naphthol	2
70	4,4'-Methyl-6,6'-chlorothiindigo	1
71	2-(Monobromo-2-oxo-1(2)-acenaphthylidene-3(2)-thianaphthenone	1
72.	α -Naphthylamine- β -naphthol	3, 4, 6, 8
73	β -Naphthylamine- β -naphthol	3, 4, 6
74	1-(1-Naphthylazo)-2-naphthol	1
75	1-(2-Naphthylazo)-2-naphthol	1
76	1-Nitro-acridone	1, 3
77.	1-(2-Nitro-o-tolylazo)-2-naphthol	1, 4
78	2-(2-Oxo-1(2)-acenaphthylidene-3(2)-thianaphthenone	1
79.	Paranitroaniline	1, 3, 13
80.	Paranitroaniline-diethylaniline	3
81.	Para Red Toner	7
82.	Paratoner Red	1, 3, 14
82	Paratoner RT 435D	1

* See Footnote at end of table

Table 1. (Contd)

Dye		Reference
84. 1-(4-Phenethyl phenylazo)-2-naphthol		1
85. p-Phenetidine-1-hydroxy naphthalene-4-sulfonic acid		3
86. p-Phenetidine- β -naphthol		3, 6
87. p-Phenylazo aniline hydrochloride		1
88. 4-Phenylazo-3-hydroxy-2-naphthoic acid	Calcolake Brilliant Red R	1
89. 1-(4-Phenylazo phenylazo)-2-naphthol	Sudan III, Oil Red, CI 248	1, 4
90. Pyronine G	*CI 45005 (Basic Dye) ⁸	4
91. Rhodamine	Rhodamine B, Rhodamine BEx CI 749	1, 4, 14, 6 10, 12, 15
92. Rhodamine 5GDN base		3
93. Rhodamine 6GDNEEx		3, 4, 10
94. Rosinduline		3
95. Rosindone	True Rosindone 7-Phenyl-5-benzo (a) phenazinone	1, 3, 9
96. Safranine T	Mixture of 3,7-diamino-2,8-dimethyl-5-phenyl-phenazinium chloride and 3,7-diamino-2,8-dimethyl-5-O-tolylphenazinium chloride CI 841, Made by DuPont	1
97. Sodium salt of o-carboxylic acid of benzene-azo- β -naphthol-3:6-disulphonic acid		3
98. Sodium salt of 4-sulpho-A-naphthalene-azo- β -naphthol-6-sulphonic acid		3
99. Sudan IV	Spirit Red III	1, 4, 6, 12
100. Tetrazotized benzidine- β -naphthol		6
101. Tetrazotized o-dianisidine- α -naphthol		6
102. Tetrazotized o-tolidine- α -naphthol		6
103. Tetrazotized o-tolidine- β -naphthol		6
104. Thioindigo	Durindone Red B, CI 1207 *CI 73300 (Vat Red 41) ⁸	1, 6
105. Xylylidene- β -naphthol		3, 6
106. 1-Xylylazo-2-naphthol	Calro Oil Scarlet II, National Oil Scarlet 6G, Sudan II, Sudan Red *CI 12140 (Solvent Orange 7) ⁸	1

* These are the new Colour Index numbers as best as can be ascertained from the information available.

Table 2. US Food and Drug Administration Dyes

Dye	Colour index number
1. FD&C #40	16035
2. FD&C #3	45430
3. Citrus Red #2	12156
4. FD&C Red #4	14700
5. D&C Red #17	26100
6. D&C Red #31	15800:1
7. D&C Red #34	15880:1
8. D&C Red #39	13058

4. DISCUSSION OF RESULTS

Investigators^{1,4,14} generally agree upon the following properties of dyes suitable for the production of colored smokes:

- a. The molecular weight must not exceed 450.
- b. The dye should be a member of one of the following series: anthraquinone, azine, azo, quinoline, or xanthene.
- c. The following groups must be absent: sulfonic, hydrochloride, nitro, nitroso, quaternary ammonium, and oxonium.
- d. The following groups may be present: amino and substituted amino, alkyl, aryl, chloro, bromo, hydroxy, and alkoxy.
- e. The dye must not have a tendency to undergo auto condensation. Of the dyes reviewed, the following have these properties:
 - (1) Rhodamine B
 - (2) Sudan IV
 - (3) 1 Xylylazo-2-naphthol
 - (4) Citrus Red #2 (2,5 dimethoxyphenylazo-2-naphthol)
 - (5) 9-Diethylaminorosindone
 - (6) O-Methoxyphenylazo-B-naphthol

The first four dyes in the list have been shown to be mutagenic or carcinogenic in studies reviewed in the Registry of Toxic Effects of Chemical Substances.¹⁶ In addition, Rhodamine B was listed as a potential health hazard in a report by Anson and Parent,¹⁵ and Citrus Red tends to flame and decompose when disseminated. For these reasons, these dyes were eliminated from further consideration.

The remaining two dyes, 9-diethylaminorosindone and o-methoxyphenylazo- β -naphthol, have been recommended by various investigators as replacement dyes for 1-N methylaminoanthraquinone.

4.1 9-Diethylaminorosindone.

Until 1960, 9-diethylaminorosindone, a member of the azine class of dyes was one of the dyes specified for use in the M18 colored-smoke hand grenade. The military specification for this dye was MIL-D-3614¹⁹ which replaced Army 96-111-100. According to Munro and Guion,⁹ 9-diethylaminorosindone produced a red smoke comparable in quality and color to that produced by 1-N methylaminoanthraquinone. Difficulties in obtaining the necessary starting materials and the difficult synthesis for the rosindone dyes as compared to the anthraquinone dyes led to the phasing out of rosindone dyes in favor of the anthraquinone dyes by the dye industry.

No present commercial supplier for 9-diethylaminorosindone was located during this study; however, a former manufacturer, Buffalo Color and Chemical Corporation (National Aniline), located in their files a procedure for synthesizing the dye. However, interest in resuming manufacture of the dye was not officially expressed by the company.

No toxicological data¹⁵⁻¹⁸ were located for this dye.

4.2 O-Methoxyphenylazo-B-naphthol.

O-Methoxyphenylazo- β -naphthol, a member of the azo class of dyes, has also been specified for use in pyrotechnics (MIL-D-3719) and was used during World War II by the Germans and Italians in pyrotechnic formulations.¹²

A thorough study of this dye by Guion² recommended that the dye be used as a replacement only if 1-N-methylaminoanthraquinone or 9-diethylaminorosindone could not be used. The smoke produced by this dye was considered by Guion² not of the same quality or hue as that produced by 1-N methylaminoanthraquinone. In addition, the O-methoxyphenylazo-B-naphthol had a tendency to flame more readily when disseminated.

A commercial supplier for this dye, was located during the study. BASF Wyandotte Corporation, markets o-methoxyphenylazo- β -naphthol under the name of Sudan Red G.

One report¹⁸ listed this dye as carcinogen, based on unpublished data which could not be corroborated. No other toxicological data were found for this dye.

5. CONCLUSIONS AND RECOMMENDATIONS

Two of the 114 dyes reviewed for this study are recommended as candidates for replacement of 1-N methylaminoanthraquinone. The dyes are 9-diethylaminorosindone and o-methoxyphenylazo- β -naphthol.

These dyes have been tested and used in pyrotechnics and produce acceptable red smoke. There are insufficient toxicological data at this time to assess the health effects of these dyes.

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